Form PTO-1390US DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE
(Rev. 5-93)

TRANSMITTAL LETTER TO THE UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US) CONCERNING A FILING UNDER 35 U.S.C. 371

ATTORNEY'S DOCKET NO. **H 4124 PCT/US**

U.S. APPLICATION NO. (if known sec. 17 CFR 1.5)
10/018274

INTERNATIONAL APPLICATION NO.	INTERNATIONAL FILING DATE	PRIORITY DATE CLAIMED
PCT/EP00/03659	**April 22, 2000**	**April 30, 1999**

TITLE OF INVENTION
USE OF NANOSCALAR ANTIMICROBIAL ACTIVE INGREDIENTS IN BODY DEODORANTS

APPLICANT(S) FOR DO/EO/US
Christine Schroeder, Hans-Theo Leinen, Bernhard Banowski, Marcel Roth, Johann Glasl

Applicant herewith submits to the United States Designated/Elected Office (EO/DO/US) the following items and other information:

1. ■ This is a **FIRST** submission of items concerning a filing under 35 U.S.C. 371.

2. ☐ This a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. 371.

3. ☐ This express request to begin national examination procedures (35 U.S.C. 371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and PCT Articles 22 and 39 (1).

4. ■ A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date.

5. ■ A copy of the International Application as filed (35 U.S. C. 371(c)(2)).
   a. ☐ is transmitted herewith (required only if not transmitted by the International Bureau).
   b. ■ has been transmitted by the International Bureau.
   c. ☐ is not required, as the application was filed in the United States Receiving Office (RO/US).

6. ■ A translation of the International Application into English (35 U.S.C. 371(c)(2)).

7. ■ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3))
   a. ☐ are transmitted herewith (required only if not transmitted by the International Bureau).
   b. ☐ have been transmitted by the International Bureau.
   c. ☐ have not been made; however, the time limit for making such amendments has NOT expired.
   d. ■ have not been made and will not be made.

8. ☐ A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).

9. ■ An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)). **UNEXECUTED**

10. ☐ A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).

Items 11. to 16. below concern other document(s) or information included:

11. ☐ An Information Disclosure Statement under 37 CFR 1.97 and 1.98.

12. ☐ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.

13. ■ A FIRST preliminary amendment
    ☐ A SECOND or SUBSEQUENT preliminary amendment.

14. ☐ A substitute specification.

15. ☐ A change of power of attorney and/or address letter.

16. ☐ Other items or information.:

"Express Mail" mailing label number EL615775499US

10/018274 06 1002

JC05 Rec'd PCT/PTO 30 OCT 2001

U.S. Application No. (If known, see 37 CFR 1.5) 10/018274	INTERNATIONAL APPLICATION NO. PCT/EP00/03659	ATTORNEY'S DOCKET NUMBER H 4124 PCT/US

17. ■ The following fees are submitted:	CALCULATIONS	PTO USE ONLY
Basic National Fee (37 CFR 1.492(a)(1)-(5)): Search Report has been prepared by the EPO or JPO.....**$890.00**		
International preliminary examination fee paid to USPTO (37CFR 1.482) **$710.00**		
No international preliminary examination fee paid to USPTO (37 CFR 1.482) but international search fee paid to USPTO (37CFR 1.445(a)(2))....**$740.00**		
Neither international preliminary examination fee (37CFR 1.482) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO. **$1,040.00**		
International preliminary examination fee paid to USPTO (37CFR 1.482) and all claims satisfied provisions of PCT Article 33(2)-(4)..... **$100.00**		
ENTER APPROPRIATE BASIC FEE AMOUNT =	$ 890	00
Surcharge of $130.00 for furnishing the oath or declaration later than ☐ 20 ☐ 30 months from the earliest claimed priority date 37 (CFR 1.492(e)).	$	

Claims	Number filed	Number Extra	Rate		
Total Claims	1 - 20 =	0	X 18.00	$	00
Independent Claims	1 - 3 =	0	X 84.00	$	00
Multiple dependent claims (s)(if applicable)		0	+ 280.00	$	00
TOTAL OF ABOVE CALCULATIONS ..... =				$ 890	00
Reduction by ½ for filing by small entity, if applicable. Verified Small Entity statement must also be filed. (Note 37 CFR 1.9, 1.27, 1.28).				$	
SUBTOTAL =				$ 890	00
Processing fee of **$130.00** for furnishing the English translation later the ☐ 20 ☐ 30 months from the earliest claimed priority date (37CFR 1.492(f)). +				$	
TOTAL NATIONAL FEE =				$ 890	00
Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). $40.00 per property +				$	
TOTAL FEES ENCLOSED =				$ 890	00
				Amount to be: refunded	$----------
				charged	890.00

a. ☐ A check in the amount of $_____ to cover the above fees is enclosed.

b. ■ Please charge my Deposit Account No. **01-1250** in the amount of **$ 890.00** to cover the above fees. A triplicate copy of this sheet is enclosed. Order No. **01-0764**.

c. ■ The Assistant Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. **01-1250**. A triplicate copy of this sheet is enclosed.

NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137 (a) or (b)) must be filed and granted to restore the application to pending status.

SEND ALL CORRESPONDENCE TO: Henkel Corporation, Law Dept.
2500 Renaissance Blvd., Ste. 200
Gulph Mills, PA 19406.....

Kimberly R. Hild
SIGNATURE

Kimberly R. Hild
NAME ATTORNEY FOR APPLICANT

39,224
REGISTRATION NUMBER

Form PTO 1390 (REV 5-93)

PATENT
Docket No. H 4124 PCT/US

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:
Schroeder, et al.

International Application No. PCT/EP00/03659
International Filing Date: April 22, 2000

Serial No. To be assigned
Filed: To be assigned

Examiner: To be assigned
Art Unit: To be assigned

Title: USE OF NANOSCALAR ANTIMICROBIAL ACTIVE INGREDIENTS IN BODY DEODORANTS

"Express Mail Post Office to Addressee" service mailing label number EL615775499US

PRELIMINARY AMENDMENT

Box PCT
Assistant Commissioner for Patents
Washington, DC 20231

Attn: DO/EO/US

Sir:

Prior to examining this application, please amend the application as follows:

In the Specification (Using the English Translation):

On page 1 of the English translation, on a separate line between the title and line 1, please insert the following paragraph:

Docket No. H4124 PCT/US
PCT/EP00/03659

-- CROSS REFERENCE TO RELATED APPLICATIONS

This application is a national stage application under 35 U.S.C. § 371 of international application PCT/EP00/03659 filed on April 22, 2000, the international application not being published in English. This application also claims priority under 35 U.S.C. §119 to DE 199 19 769.5 filed on April 30, 1999.

In the Claims

Please cancel Claims 2 to 11, without prejudice.

Docket No. H4124 PCT/US
PCT/EP00/03659

REMARKS

Applicants respectfully request the Examiner to enter the above amendments prior to examination of this application.

Status of Claims

Claim 1 will be pending after entry of the present amendment. Claims 2 to 11 are being canceled without prejudice.

Amendment

The specification is being amended to insert a cross-reference to related applications in accordance 37 CFR §1.78 and to claim priority to those applications listed therein.

No new matter is added by the new claims or amendments to the specification.

CONCLUSION

Applicants respectfully request early and favorable notification of allowance of all pending claims. The Assistant Commissioner is authorized to charge any deficiency in the required fee or to credit any overpayment to Deposit Account 01-1250 in connection with this amendment.

Respectfully submitted,

Kimberly R. Hild

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"Express Mail " Mailing Label Number EL780369825US .

PATENT
Docket No. C 2583 PCT/US

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re: Application of Schroeder, et al.

Serial No. 10/018,274 Examiner: To be assigned
Filed: 06/10/2002 Art Unit: To be assigned
PCT/EP00/03659
International Filing Date: April 22, 2000
Priority Date Claimed: April 30, 1999
TITLE: USE OF NANOSCALAR ANTIMICROBIAL ACTIVE INGREDIENTS IN BODY DEODORANTS

SUPPLEMENTAL PRELIMINARY AMENDMENT

Commissioner for Patents
Box PCT
Washington, DC 20231

ATTN: DO/EO/US

Sir:

Prior to examining this application, please amend the application as follows:

In the Specification (Using the English Translation):

On page 1, on a separate line after the paragraph entitled "Cross Reference to Related Applications" (added in the preliminary amendment filed on October 30, 2001) and before line 1 of the English translation, please insert the following header:
-- BACKGROUND OF THE INVENTION -- .

On page 2, please replace the paragraph beginning at line 14 and ending on line 21 with the following new paragraph:

-- Because of their physiochemical properties and their own odors, the active principles used in body deodorants are often attended in practice by the problem that they only be incorporated in deodorant formulations with difficulty or in inadequate concentrations so that the

formulations obtained show unsatisfactory antimicrobial activity. In addition, there is a demand among consumers for body deodorants which work with reduced concentrations of the active principle without any loss of deodorizing effect and hence offer physiological, economic and/or ecological advantages. --

On page 3, on a separate line between lines 3 and 4, please insert the following header: --SUMMARY OF THE INVENTION--

On page 3, on a separate line between lines 27 and 28, please insert the following header: --DETAILED DESCRIPTION OF THE INVENTION--

On page 13, please replace the paragraph beginning at line 27 and ending on line 29 with the following new paragraph:

--To produce the body deodorants according to the invention, the nanoscale antimicrobial agents are mixed with the other formulation ingredients in known manner.--

On page 16, line 1, please delete the heading "CLAIMS" and insert therefor: --What is claimed is:--

On a separate page, after page 17, please insert the enclosed Abstract of the Disclosure.

In the claims:

Please cancel claim 1, without prejudice.

Please add the following new claims:

12. (New) A method of making a body deodorant comprising forming a deodorant composition comprising nanoscale antimicrobial particles wherein the nanoscale antimicrobial particles comprise one or more antimicrobial agents and have a particle diameter in the range of

from about 5 nanometers to about 500 nanometers.

13. (New) The method of claim 12 wherein the antimicrobial agents are active against gram-positive bacteria.

14. (New) The method of claim 13 wherein the antimicrobial agents are active against Corynebacterium xerosis.

15. (New) The method of claim 14 wherein the antimicrobial agents comprise salicylic acid-n-octyl amide, or salicylic acid-n-decyl amide, or combinations thereof.

16. (New) The method of claim 14 wherein the antimicrobial agents comprise 2,4,4'-trichloro-2'-hydroxydiphenyl ether.

17. (New) The method of claim 12 wherein the nanoscale antimicrobial particles are obtained by a process comprising:

(a) adding the antimicrobial agents into a liquid phase to form a liquid mixture, wherein the antimicrobial agents are insoluble in the liquid phase;

(b) heating the liquid mixture to at least a temperature beyond the melting point of the antimicrobial agents;

(c) adding an effective quantity of at least one emulsifier to the liquid mixture to form an emulsion; and

(d) cooling the emulsion to below the melting point of the antimicrobial agents.

18. (New) The method of claim 12 wherein the antimicrobial agents comprise an antimicrobial perfume.

19. (New) The method of claim 12 wherein the antimicrobial agents comprise salicylic acid-n-octyl amide, or salicylic acid-n-decyl amide, or combinations thereof.

20. (New) The method of claim 12 wherein the nanoscale antimicrobial particles are coated with a coating comprising one or more emulsifiers, or protective colloids, or mixtures thereof.

21. (New) The method of claim 12 wherein the nanoscale antimicrobial particles comprise from about 0.01 wt% to about 5 wt% of the antimicrobial agents based on the total weight of the nanoscale particles.

22. (New) The method of claim 12 wherein the deodorant composition is in the form of a deodorizing aerosol, pump spray, roll-on preparation, or stick preparation.

23. (New) A body deodorant composition comprising nanoscale antimicrobial particles wherein the nanoscale antimicrobial particles comprise one or more antimicrobial agents and have a particle diameter in the range of from about 5 nanometers to about 500 nanometers.

24. (New) The composition of claim 23 wherein the antimicrobial agents are active against Corynebacterium xerosis.

25. (New) The composition of claim 24 wherein the nanoscale antimicrobial particles comprise from about 0.01 wt% to about 5 wt% of the antimicrobial agents based on the total weight of the nanoscale particles.

26. (New) The composition of claim 25 wherein the deodorant composition is in the form of a deodorizing aerosol, pump spray, roll-on preparation, or stick preparation.

27. (New) The composition of claim 26 wherein the antimicrobial agents comprise salicylic acid-n-octyl amide, or salicylic acid-n-decyl amide, or combinations thereof.

28. (New) The composition of claim 26 wherein the antimicrobial agents comprise 2,4,4'-trichloro-2'-hydroxydiphenyl ether.

29. (New) A method of preventing or treating body odors comprising:

(a) providing a deodorant composition comprising nanoscale antimicrobial particles wherein the nanoscale antimicrobial particles comprise one or more antimicrobial agents and have a particle diameter in the range of from about 5 nanometers to about 500 nanometers; and

(b) applying the deodorant composition to a body.

30. (New) The method of claim 29 wherein the nanoscale antimicrobial particles comprise from about 0.01 wt% to about 5 wt% of the antimicrobial agents based on the total weight of the nanoscale particles.

31. (New) The method of claim 30 wherein the deodorant composition is in the form of a deodorizing aerosol, pump spray, roll-on preparation, or stick preparation.

32. (New) The method of claim 30 wherein the antimicrobial agents comprise salicylic acid-n-octyl amide, or salicylic acid-n-decyl amide, or combinations thereof.

Case C 2583 PCT/US
Serial No. 10/018,274

REMARKS

Applicants respectfully request the Examiner to enter the above amendments prior to examination of this application.

Status of Claims

Claims 12 to 32 will be pending after entry of the present amendment. Claim 1 is being canceled without prejudice and Claims 12 to 32 are being added.

Amendment

The specification is also being amended to correct typographical errors at pages 2 and 13 of the specification. These error would have been readily apparent to one skilled in the art.

The Specification has been amended to include the preferred section headings pursuant to 37 C.F.R. §1. 77(b). It is submitted that the amendments to the Specification made herein introduce no new matter. Their entry is therefore proper and respectfully requested. An Abstract of the Disclosure has been added on a separate sheet following the claims.

New claims 12 to 32 are being presented solely for the purpose of improving clarity and grammar, which may suffer in translation, and not for any reason related to the statutory requirements for a patent. New claims 12 to 32 have not been added in response to any rejection, or in anticipation of any rejection related to the statutory requirements for a patent. New claims 12 to 32 are supported by the specification and no new matter has been introduced. Entry is therefore proper and respectfully requested. Prompt examination of the instant application in view of the amendments made herein is respectfully requested.

Respectfully submitted,

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ABSTRACT OF THE DISCLOSURE

A deodorant composition and method of making and using a deodorant composition are provided. The deodorant composition contains nanoscale antimicrobial particles where the nanoscale antimicrobial particles contain one or more antimicrobial agents and have a particle diameter in the range of from 5 nanometers to 500 nanometers.

Use of Nanoscalar Antimicrobial Active Ingredients in Body Deodorants

This invention relates to the use of antimicrobial agents in nanoscale form for the production of body deodorants.

Body deodorants, also known simply as deodorants, are formulations which counteract, mask or eliminate body odors. Body odors are formed by the action of skin bacteria on apocrine perspiration which results in the formation of unpleasant-smelling degradation products. Accordingly, deodorants contain active principles which act as antibacterial agents, enzyme inhibitors, odor absorbers or odor maskers.

Nanoscale materials are materials whose particle diameter in the direction of the largest dimension of the particles is less than 1000 nm (nanometers). In the present specification, the term "nanoparticulate" is used synonymously with the term "nanoscale". Nanoscale active principles are described in the literature in particular as agents for achieving a controlled release of the active principle over a prolonged period. For example, **WO 98/14174** describes nanoparticles for parenteral therapeutic use which consist of a pharmacologically active substance encapsulated in a shell of a biodegradable polymer. The document in question mentions inter alia antibacterial agents, such as chloramphenicol and vanomycin, and antimicrobial agents, such as penicillins and cephalosporins, as examples of pharmacologically active substances. Antimicrobial products containing nanoscale Schiff's bases of aromatic aldehydes are known from **DE 4402103** which describes the use of these products for the lasting antimicrobial finishing of textiles. Patent application **CA 2,111,523** describes disinfectants which, besides other constituents, also contain surface-modified nanoparticulate antimicrobial agents. A disinfecting cleaner formulation is mentioned as an example. Patent application **CA**

2,111,522 describes compositions with a long-lasting germicidal effect which contain surface-modified nanoparticulate antimicrobial agents. Disinfectants for surface treatment which form permanent antimicrobial films on the treated surface are mentioned as applications of these compositions. However, there is nothing in the prior art to suggest that nanoparticulate antimicrobial agents can be used with advantage as active principles in body deodorants. Although it is known to the expert that antimicrobial agents are used, for example, both in surface disinfection and in body deodorants, the expert also knows that the form of application and the requirements in regard to strength of effect, action spectrum and the formulation of the active principles are so different in the various fields of application that the knowledge acquired in one field of application cannot obviously be applied to another field of application.

Because of their physicochemical properties and their own odors, the active principles used in body deodorants are often attended in practice by the problem that they only be incorporated in deodorant formulations with difficulty or in inadequate concentrations so that the formulations obtained show unsatisfactory antimicrobial activity. In addition, there is a demand among consumers for body deodorants which work with reduced concentrations of the active principle without any loss of deodorizing effect and hence offer physiological, economic and/or ecological advantages.

Accordingly, one problem addressed by the present invention was to enable body deodorants to be produced using antimicrobial agents which, due for example to their poor solubility or their strong odor, can only be conventionally incorporated in body deodorants with difficulty or in inadequate concentrations.

Another problem addressed by the invention was to provide body deodorants with sufficient antimicrobial activity for practical application and, at the same time, a reduced content of antimicrobial agents.

The problems stated above have been solved by the use of the

antimicrobial agents in the form of nanoparticles with a particle diameter of 5 to 500 nm and preferably 10 to 150 nm for the production of body deodorants.

In a first embodiment, therefore, the present invention relates to the use of nanoscale antimicrobial agents with a particle diameter of 5 to 500 nm and preferably 10 to 150 nm for the production of body deodorants, more especially deodorizing aerosols, pump sprays, roll-ons and sticks. The use of the nanoscale antimicrobial agents is particularly suitable for the production of products which are required to show only bacteriostatic activity and not bactericidal activity.

It has surprisingly been found that the following advantages, for example, are achieved in this way:

a) The incorporation of antimicrobial agents in deodorant formulations is improved to the extent that lipophilic active principles can be incorporated more easily in aqueous formulations while hydrophilic active principles can be incorporated more easily in nonaqueous or low-water formulations.

b) the effectiveness of the active principles from the formulations is increased. This means that, for the same quantity by weight, the nanoparticulate active principle has a stronger antimicrobial effect than the same active principle in a larger particle size corresponding to the prior art.

c) In the case of active principles with a strong odor of their own, the odor can be weakened or even suppressed by surface modification of the nanoscale particles.

Antibacterial agents with substantially selective activity against bacteria involved in the formation of odor-generating substances in bodily perspiration are particularly suitable for the use according to the invention.

Where antimicrobial agents are used, it is important to ensure that the population of the bacteria concerned is merely controlled to prevent excessive growth (bacteriostatic effect) and not to destroy the bacteria completely (which would correspond to bactericidal activity).

Any substances active against gram-positive bacteria are particularly suitable as antimicrobial agents according to the invention. Substances active against Corynebacterium xerosis are particularly preferred. The active substances according to the invention include, for example,

- 4-hydroxybenzoic acid, its salts with alkali or alkaline earth metals or its esters with linear or branched C_{1-10} alcohols,
- N-(4-chlorophenyl)-N'-(3,4-dichlorophenyl)-urea,
- 2,4,4'-trichloro-2'-hydroxy diphenyl ether (triclosan),
- 4-chloro-3,5-dimethyl phenol,
- 2,2'-methylene-bis-(6-bromo-4-chlorophenol),
- 3-methyl-4-(1-methylethyl)-phenol,
- 2-benzyl-4-chlorophenol,
- 3-(4-chloropenoxy)-propane-1,2-diol,
- 3-iodo-2-propinyl butyl carbamate,
- chlorohexidine,
- 3,4,4'-trichlorocarbanilide (TTC),
- 1-hydroxy-4-methyl-6-(2,4,4-trimethylpentyl)-2-(1H)-pyridinone, ethanolamine salt (1:1) (Octopirox)
- antimicrobial perfumes such as, for example, thymol or menthol,
- glycerol monolaurate (GML),
- diglycerol monocaprate (DMC),
- zinc salts such as, for example, zinc glycinate, zinc lactate or zinc phenol sulfonate,
- phytosphingosines,
- dodecane-1,2-diol,

- undecylenic acid, its salts with alkali or alkaline earth metals or its esters with linear or branched C_{1-10} alcohols,
- salicylic acid-N-alkyl amides where the alkyl groups contain 1 to 22 carbon atoms and may be linear or branched

and mixtures thereof.

Particularly preferred antimicrobial agents according to the invention are salicylic acid-N-octyl amide and/or salicylic acid-N-decyl amide, 2,4,4'-trichloro-2'-hydroxydiphenyl ether and antimicrobially active perfumes.

The nanoscale active principles consist of a discrete phase of the active principle with preferably at least one surface modifier adsorbed onto its surface. Particularly suitable surface modifiers are emulsifiers and/or protective colloids. The coating of the particles with emulsifiers and/or protective colloids prevents subsequent agglomeration of the particles.

Suitable emulsifiers are, for example, nonionic surfactants from at least one of the following groups:

(1) products of the addition of 2 to 30 moles of ethylene oxide and/or 0 to 5 moles of propylene oxide onto linear C_{8-22} fatty alcohols, C_{12-22} fatty acids and alkyl phenols containing 8 to 15 carbon atoms in the alkyl group;

(2) $C_{12/18}$ fatty acid monoesters and diesters of addition products of 1 to 30 moles of ethylene oxide onto glycerol;

(3) glycerol monoesters and diesters and sorbitan monoesters and diesters of saturated and unsaturated fatty acids containing 6 to 22 carbon atoms and ethylene oxide adducts thereof;

(4) alkyl mono- and oligoglycosides containing 8 to 22 carbon atoms in the alkyl group and ethoxylated analogs thereof;

(5) products of the addition of 15 to 60 moles of ethylene oxide onto castor oil and/or hydrogenated castor oil;

(6) polyol esters and, in particular, polyglycerol esters such as, for

example, polyglycerol polyricinoleate, polyglycerol poly-12-hydroxystearate or polyglycerol dimerate. Mixtures of compounds from several of these classes are also suitable;

(7) products of the addition of 2 to 15 moles of ethylene oxide onto castor oil and/or hydrogenated castor oil;

(8) partial esters based on linear, branched, unsaturated or saturated $C_{6/22}$ fatty acids, ricinoleic acid and 12-hydroxystearic acid and glycerol, polyglycerol, pentaerythritol, dipentaerythritol, sugar alcohols (for example sorbitol), sucrose, alkyl glucosides (for example methyl glucoside, butyl glucoside, lauryl glucoside) and polyglucosides (for example cellulose);

(9) mono-, di and trialkyl phosphates and mono-, di- and/or tri-PEG-alkyl phosphates and salts thereof;

(10) wool wax alcohols;

(11) polysiloxane/polyalkyl polyether copolymers and corresponding derivatives;

(12) mixed esters of pentaerythritol, fatty acids, citric acid and fatty alcohol according to **DE-PS 11 65 574** and/or mixed esters of fatty acids containing 6 to 22 carbon atoms, methyl glucose and polyols, preferably glycerol or polyglycerol, and

(13) polyalkylene glycols.

The addition products of ethylene oxide and/or propylene oxide onto fatty alcohols, fatty acids, alkylphenols, glycerol monoesters and diesters and sorbitan monoesters and diesters of fatty acids or with castor oil are known commercially available products. They are homolog mixtures of which the average degree of alkoxylation corresponds to the ratio between the quantities of ethylene oxide and/or propylene oxide and substrate with which the addition reaction is carried out. $C_{12/18}$ fatty acid monoesters and diesters of addition products of ethylene oxide onto glycerol are known as

refatting agents for cosmetic formulations from **DE-PS 20 24 051**.

$C_{8/18}$ alkyl mono- and oligoglycosides, their production and their use are known from the prior-art literature. They are produced in particular by reacting glucose or oligosaccharides with primary alcohols containing 8 to 18 carbon atoms. So far as the glycoside component is concerned, both monoglycosides where a cyclic sugar unit is attached to the fatty alcohol by a glycoside bond and oligomeric glycosides with a degree of oligomerization of preferably up to about 8 are suitable. The degree of oligomerization is a statistical mean value on which a homolog distribution typical of such technical products is based.

Typical examples of anionic emulsifiers are soaps, alkyl benzene-sulfonates, alkanesulfonates, olefin sulfonates, alkylether sulfonates, glycerol ether sulfonates, α-methyl ester sulfonates, sulfofatty acids, alkyl sulfates, fatty alcohol ether sulfates, glycerol ether sulfates, hydroxy mixed ether sulfates, monoglyceride (ether) sulfates, fatty acid amide (ether) sulfates, mono- and dialkyl sulfosuccinates, mono- and dialkyl sulfosuccinamates, sulfotriglycerides, amide soaps, ether carboxylic acids and salts thereof, fatty acid isethionates, fatty acid sarcosinates, fatty acid taurides, N-acylamino acids such as, for example, acyl lactylates, acyl tartrates, acyl glutamates and acyl aspartates, alkyl oligoglucoside sulfates, protein fatty acid condensates (particularly wheat-based vegetable products) and alkyl - (ether) phosphates. If the anionic surfactants contain polyglycol ether chains, they may have a conventional homolog distribution although they preferably have a narrow-range homolog distribution.

Other suitable emulsifiers are zwitterionic surfactants. Zwitterionic surfactants are surface-active compounds which contain at least one quaternary ammonium group and at least one carboxylate and one sulfonate group in the molecule. Particularly suitable zwitterionic surfactants are the so-called betaines, such as the N-alkyl-N,N-dimethyl ammonium glycinates, for example cocoalkyl dimethyl ammonium

glycinate, N-acylaminopropyl-N,N-dimethyl ammonium glycinates, for example cocoacylaminopropyl dimethyl ammonium glycinate, and 2-alkyl-3-carboxymethyl-3-hydroxyethyl imidazolines containing 8 to 18 carbon atoms in the alkyl or acyl group and cocoacylaminoethyl hydroxyethyl carboxymethyl glycinate. The fatty acid amide derivative known under the CTFA name of *Cocamidopropyl Betaine* is particularly preferred. Ampholytic surfactants are also suitable emulsifiers. Ampholytic surfactants are surface-active compounds which, in addition to a $C_{8/18}$ alkyl or acyl group, contain at least one free amino group and at least one -COOH- or -SO_3H- group in the molecule and which are capable of forming inner salts. Examples of suitable ampholytic surfactants are N-alkyl glycines, N-alkyl propionic acids, N-alkylaminobutyric acids, N-alkyliminodipropionic acids, N-hydroxyethyl-N-alkylamidopropyl glycines, N-alkyl taurines, N-alkyl sarcosines, 2-alkylaminopropionic acids and alkylaminoacetic acids containing around 8 to 18 carbon atoms in the alkyl group. Particularly preferred ampholytic surfactants are N-cocoalkylaminopropionate, cocoacylaminoethyl aminopropionate and $C_{12/18}$ acyl sarcosine. According to the invention, other suitable emulsifiers besides ampholytic surfactants are quaternary emulsifiers, those of the esterquat type, preferably methyl-quaternized difatty acid triethanolamine ester salts, being particularly preferred. Typical examples of anionic emulsifiers are are alkyl sulfates, alkyl ether sulfates and monoglyceride (ether) sulfates.

In general, the active principles and the emulsifiers are used in a ratio by weight of 1:100 to 100:1, preferably 1:25 to 25:1 and more preferably 1:10 to 10:1. Emulsifiers capable of forming microemulsions are particularly preferred.

Suitable protective colloids are, for example, gelatine, casein, gum arabic, lysalbinic acid, starch, carboxymethyl cellulose or modified carboxymethyl cellulose and polymers such as, for example, polyvinyl alcohols, polyvinyl pyrrolidones, polyalkylene glycols and polyacrylates.

Accordingly, the present invention also relates to the use according to the invention of nanoscale antimicrobial agents where the nanoparticles are coated with one or more emulsifiers and/or protective colloids.

The nanoparticles according to the invention can be produced, for example, by

(a) introducing active principles into a liquid phase in which they are insoluble,

(b) heating the resulting mixture to beyond the melting point of the active principles,

(c) adding an effective quantity of at least one emulsifier to the resulting oil phase and finally

(d) cooling the emulsion to below the melting point of the active principles.

Accordingly, the present invention also relates to the use according to the invention of nanoscale antimicrobial agents produced by this process.

Another process for the production of nanoparticles by rapid expansion of supercritical solutions (RESS) is known from the article by S. Chihlar, M. Türk and K. Schaber in **Proceedings World Congress on Particle Technology 3, Brighton, 1998**. To prevent the nanoparticles from re-agglomerating, it is advisable to dissolve the starting materials in the presence of suitable protective colloids or emulsifiers and/or to expand the critical solutions into aqueous and/or alcoholic solutions of the protective colloids or emulsifiers or into cosmetic oils which may in turn contain redissolved emulsifiers and/or protective colloids.

Another suitable process for the production of nanoscale particles is the **evaporation technique**. Here, the starting materials are first dissolved in a suitable organic solvent (for example alkanes, vegetable oils, ethers,

esters, ketones, acetals and the like). The resulting solutions are then introduced into water or another non-solvent, generally in the presence of a surface-active compound dissolved therein, in such a way that the nanoparticles are precipitated by the homogenization of the two immiscible solvents, the organic solvent preferably evaporating. O/w emulsions or o/w microemulsions may be used instead of an aqueous solution. The emulsifiers and protective colloids mentioned at the beginning may be used as the surface-active compounds. Another method for the production of nanoparticles is the so-called GAS process (gas anti-solvent recrystallization). This process uses a highly compressed gas or supercritical fluid (for example carbon dioxide) as non-solvent for the crystallization of dissolved substances. The compressed gas phase is introduced into the primary solution of the starting materials and absorbed therein so that there is an increase in the liquid volume and a reduction in solubility and fine particles are precipitated. The PCA process (precipitation with a compressed fluid anti-solvent) is equally suitable. In this process, the primary solution of the starting materials is introduced into a supercritical fluid which results in the formation of very fine droplets in which diffusion processes take place so that very fine particles are precipitated. In the PGSS process (particles from gas saturated solutions), the starting materials are melted by the introduction of gas under pressure (for example carbon dioxide or propane). Temperature and pressure reach near- or super-critical conditions. The gas phase dissolves in the solid and lowers the melting temperature, the viscosity and the surface tension. On expansion through a nozzle, very fine particles are formed as a result of cooling effects.

The above-mentioned production processes for the nanoparticles according to the invention are merely examples and are not intended to limit the invention in any way.

The body deodorants obtainable using the nanoscale antimicrobial

agents in accordance with the invention may also contain, for example, fatty acids in the form of their alkali metal soaps, polyols, lower alcohols, enzyme inhibitors, odor absorbers, odor maskers, water, complexing agents, antioxidants, preservatives, perfumes, colorants, opacifiers, pearlizing pigments, fine-particle silica, consistency factors, gel formers, waxes, fatty alcohols, emulsifiers, thickeners and other suitable formulation bases as further auxiliaries and additives.

Fatty acids in the context of the invention are C_{16-22} carboxylic acids such as, for example, palmitic acid, stearic acid and behenic acid or technical mixtures consisting predominantly of such fatty acids, for example hydrogenated palm oil fatty acid or hydrogenated tallow fatty acid.

Polyols in the context of the invention are those containing 3 to 6 carbon atoms and 2 to 6 hydroxyl groups such as, for example, ethylene glycol, 1,2-propylene glycol, 1,3-propylene glycol, 1,2-butylene glycol, 1,3-butylene glycol, 1,4-butylene glycol, glycerol, erythritol, pentaerythritol, trimethylol propane, sorbitol, anhydrosorbitol, cyclohexane triol or inositol.

The preparations may contain ethanol or isopropanol, for example, as lower alcohols.

Suitable enzyme inhibitors are, for example, esterase inhibitors. Esterase inhibitors are preferably trialkyl citrates, such as trimethyl citrate, tripropyl citrate, triisopropyl citrate, tributyl citrate and, in particular, triethyl citrate (Hydagen® CAT, Henkel KGaA, Düsseldorf, FRG). Esterase inhibitors inhibit enzyme activity and thus reduce odor formation. Other esterase inhibitors are sterol sulfates or phosphates such as, for example, lanosterol, cholesterol, campesterol, stigmasterol and sitosterol sulfate or phosphate, dicarboxylic acids and esters thereof, for example glutaric acid, glutaric acid monoethyl ester, glutaric acid diethyl ester, adipic acid, adipic acid monoethyl ester, adipic acid diethyl ester, malonic acid and malonic acid diethyl ester, hydroxycarboxylic acids and esters thereof, for example citric acid, malic acid, tartaric acid or tartaric acid diethyl ester, and zinc

glycinate.

Suitable odor absorbers are substances which are capable of absorbing and largely retaining the odor-forming compounds. They reduce the partial pressure of the individual components and thus also reduce the rate at which they spread. An important requirement in this regard is that perfumes must remain unimpaired. Odor absorbers are not active against bacteria. They contain, for example, a complex zinc salt of ricinoleic acid or special perfumes of largely neutral odor known to the expert as "fixateurs" such as, for example, extracts of ladanum or styrax or certain abietic acid derivatives as their principal component. Odor maskers are perfumes or perfume oils which, besides their odor-masking function, impart their particular perfume note to the deodorants. Suitable perfume oils are, for example, mixtures of natural and synthetic fragrances. Natural fragrances include the extracts of blossoms, stems and leaves, fruits, fruit peel, roots, woods, herbs and grasses, needles and branches, resins and balsams. Animal raw materials, for example civet and beaver, may also be used. Typical synthetic perfume compounds are products of the ester, ether, aldehyde, ketone, alcohol and hydrocarbon type. Examples of perfume compounds of the ester type are benzyl acetate, p-tert.butyl cyclohexylacetate, linalyl acetate, phenyl ethyl acetate, linalyl benzoate, benzyl formate, allyl cyclohexyl propionate, styrallyl propionate and benzyl salicylate. Ethers include, for example, benzyl ethyl ether while aldehydes include, for example, the linear alkanals containing 8 to 18 carbon atoms, citral, citronellal, citronellyloxyacetaldehyde, cyclamen aldehyde, hydroxy-citronellal, lilial and bourgeonal. Examples of suitable ketones are the ionones and methyl cedryl ketone. Suitable alcohols are anethol, citronellol, eugenol, isoeugenol, geraniol, linalool, phenylethyl alcohol and terpineol. The hydrocarbons mainly include the terpenes and balsams. However, it is preferred to use mixtures of different perfume compounds which, together, produce an agreeable fragrance. Other suitable perfume oils are essential

oils of relatively low volatility which are mostly used as aroma components. Examples are sage oil, camomile oil, clove oil, melissa oil, mint oil, cinnamon leaf oil, lime-blossom oil, juniper berry oil, vetiver oil, olibanum oil, galbanum oil, ladanum oil and lavendin oil. The following are preferably used either individually or in the form of mixtures: bergamot oil, dihydromyrcenol, lilial, lyral, citronellol, phenylethyl alcohol, α-hexylcinnamaldehyde, geraniol, benzyl acetone, cyclamen aldehyde, linalool, Boisambrene Forte, Ambroxan, indole, hedione, sandelice, citrus oil, mandarin oil, orange oil, allylamyl glycolate, cyclovertal, lavendin oil, clary oil, β-damascone, geranium oil bourbon, cyclohexyl salicylate, Vertofix Coeur, Iso-E-Super, Fixolide NP, evernyl, iraldein gamma, phenylacetic acid, geranyl acetate, benzyl acetate, rose oxide, romillat, irotyl and floramat.

In order to be able to apply the active principles to the skin in a measurable, economic, convenient and cosmetically attractive manner, they have to be incorporated in suitable formulation bases. The most important of these are alcoholic and aqueous/alcoholic solutions, emulsions, gels, sticks - for example glycolic soap sticks - oils, wax/fat compounds and powders. Stabilizers, consistency factors and foam inhibitors, for example, may be used as additional auxiliaries.

Suitable supply forms for deodorants are aerosols, pump sprays, roll-ons, sticks and gels and also creams and powders.

The quantity in which the nanoscale compounds are used is selected so that the concentration of the antimicrobial agents present in the nanoparticles is normally between 0.01 and 5% by weight and preferably between 0.1 and 2% by weight, based on the preparations.

To produce the oral and/or dental care preparations according to the invention, the nanoscale antimicrobial agents are mixed with the other formulation ingredients in known manner.

The present invention also relates to body deodorants containing

antimicrobial agents which are characterized in that the antimicrobial agent is incorporated in the form of nanoparticles with a particle diameter of 5 to 500 nm and preferably 10 to 150 nm.

Other embodiments and/or further developments are covered by the subsidiary claims.

Examples

The following Examples are intended to illustrate the invention.

Example 1: preparation of nanoscale salicylic acid-N-octyl amide

0.5 g of salicylic acid-N-octyl amide (Mp. ca. 45°C) were dissolved in 100 g of deionized water and the mixture was heated to around 50°C, resulting in the formation of a two-phase mixture of water and amide phase. The amide phase was emulsified by addition of 8.9 g of alkyl ether sulfate (Texapon® N 70, Henkel KGaA, Düsseldorf) to form a clear mixture. The gradual passing of the oil phase into the transparent water/amide/emulsifier mixture may be taken as an indication of the formation of a microemulsion. The microemulsion was cooled to ambient temperature with continued stirring and was then concentrated by evaporation to dryness in a rotary evaporator, 9.4 g of the salicylic acid-N-octyl amide encapsulated in the ether sulfate matrix being obtained in nanoparticulate form. The nanoparticles could be reprocessed with ten times the quantity of water to form a stable and transparent dispersion. In light scattering, the particles showed a maximum with numerical weighting at a particle size of 120 nm.

Example 2: preparation of a nanoscale aqueous salicylic acid-N-octyl amide dispersion

1.0 g of salicylic acid-N-octyl amide (Mp. ca. 45°C) were emulsified with 30 g of deionized water, 30 g of Polydiol 400 (PEG-8) and 2 g of polyoxyethylene glycerol fatty acid ester (Tagat S) and slowly heated to

52°C. 30 g of fatty acid amidoalkyl betaine (Tego Betain BL 215) were then added, a clear stable dispersion being formed. The mixture was then allowed to cool to room temperature. 93 g of a transparent dispersion were obtained. In light scattering, the particles showed a maximum with numeral weighting at a particle size of 15 nm.

Example 3:

Formulation Example for a deodorizing pump spray formulation:

Ingredient	Content (% by weight)
Hydrogenated castor oil + 40 moles EO (Eumulgin HRE, Henkel KGaA)	2
Aqueous dispersion of nanoscale salicylic acid-N-octyl amide from Example 2	10
Perfume oil	0.3
Glycerol	7.7
Water	80

CLAIMS

1. The use of nanoscale antimicrobial agents with a particle diameter in the range from 5 to 500 nm for the production of body deodorants.

2. The use claimed in claim 1, characterized in that the antimicrobial agents are active against gram-positive bacteria.

3. The use claimed in claims 1 and/or 2, characterized in that the antimicrobial agents are active against Corynebacterium xerosis.

4. The use claimed in at least one of claims 1 to 3, characterized in that salicylic acid-n-octyl amide and/or salicylic acid-n-decyl amide are used as the antimicrobial agent.

5. The use claimed in at least one of claims 1 to 3, characterized in that 2,4,4'-trichloro-2'-hydroxydiphenyl ether is used as the antimicrobial agent.

6. The use claimed in at least one of claims 1 to 3, characterized in that an antimicrobial perfume is used as the antimicrobial agent.

7. The use claimed in at least one of claims 1 to 6, characterized in that nanoscale active principles obtained by

(a) introducing active principles into a liquid phase in which they are insoluble,

(b) heating the resulting mixture to beyond the melting point of the active principles,

(c) adding an effective quantity of at least one emulsifier to the resulting oil phase and finally

(d) cooling the emulsion to below the melting point of the active principles

are used.

8. The use claimed in at least one of claims 1 to 7, characterized in that nanoparticles coated with one or more emulsifiers and/or protective colloids are used.

9. The use claimed in at least one of claims 1 to 8, characterized in that the nanoscale active principles are used in such quantities that the concentration of the antimicrobial active principles present in the nanoparticles is from 0.01 to 5% by weight, based on the preparations.

10. The use claimed in at least one of claims 1 to 9, characterized in that the nanoscale active principles are used for the production of deodorizing aerosols, pump sprays, roll-ons and sticks.

11. A body deodorant containing an antimicrobial agent, characterized in that the antimicrobial agent is incorporated in the form of nanoparticles with a particle diameter of 5 to 500 nm.

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(81) Bestimmungsstaaten: AU, BR, CA, CN, CZ, HU, JP, MX, NO, PL, SK, US, europäisches Patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE).

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Vor Ablauf der für Änderungen der Ansprüche zugelassenen Frist; Veröffentlichung wird wiederholt falls Änderungen eintreffen.

(54) Title: USE OF NANOSCALAR ANTIMICROBIAL ACTIVE INGREDIENTS IN BODY DEODORANTS

(54) Bezeichnung: VERWENDUNG NANOSKALIGER ANTIMIKROBIELLER WIRKSTOFFE IN KÖRPERDEODORANTIEN

(57) Abstract

The invention relates to the use of nanoscalar antimicrobial active ingredients, with particle diameters ranging between 5 and 500 nm, for producing body deodorants. The especially fine dispersion of the particles compared to active ingredient types in prior art, facilitates the improved penetration of the active ingredients in formulations and their increased effectiveness.

(57) Zusammenfassung

Vorgeschlagen wird die Verwendung von nanoskaligen antimikrobiellen Wirkstoffen mit Teilchendurchmessern im Bereich von 5 bis 500 nm zur Herstellung von Körperdeodorantien. Gegenüber Wirkstofformen des Stands der Technik bewirkt die besondere Feinteiligkeit der Partikel eine verbesserte Einarbeitbarkeit der Wirkstoffe in Formulierungen und eine verbesserte Wirksamkeit.

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PATENT
Docket No. H 4124 PCT/US (2583 PCT/US)

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re: Application of Schroeder, et al.

Serial No. 10/018,274 Examiner: To be assigned
Filed: 06/10/02 Art Unit: To be assigned
TITLE: USE OF NANOSCALAR ANTIMICROBIAL ACTIVE INGREDIENTS IN BODY DEODORANTS

APPOINTMENT OF ASSOCIATE ATTORNEY AND/OR AGENT

Commissioner for Patents
Washington, D.C. 20231

Sir:

I, Kimberly R. Hild, hereby appoint **John E. Drach (Reg. No. 32,891)**, **Aaron R. Ettelman (Reg. No. 42,516)**, **Steven J. Trzaska (Reg. No. 36,296)** and **Henry E. Millson, Jr. (Reg. No. 18,980)** as Associate Attorney and/or Agent as provided in 37 CFR 1.34 to transact all business with the U.S. Patent and Trademarks Office in connection with the above application and any continuation, division, and/or continuation-in-part thereof, and hereby ratify any and all acts done prior to such appointment in connection with the above application.

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Respectfully submitted,

June 6, 2002
Date

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DECLARATION FOR UTILITY OR DESIGN PATENT APPLICATION

☐ Declaration Submitted with Initial Filing OR ☒ Declaration Submitted after Initial Filing

Attorney Docket Number	H 4124 PCT/US (C 2583 PCT/US)
First Named Inventor	Schroeder, Christine
COMPLETE IF KNOWN	
Application Number	10/018,274
Filing Date	06/10/2002
Group Art Unit	
Examiner Name	

As a below named inventor, I hereby declare that:
My residence, post office address, and citizenship are as stated below next to my name.
I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

USE OF NANOSCALAR ANTIMICROBIAL ACTIVE INGREDIENTS IN BODY DEODORANTS

(Title of the Invention)

the specification of which

☐ is attached hereto

OR

☒ was filed on (MM/DD/YYYY) 04/22/2000 as United States Application Number or PCT International Application Number PCT/EP00/03659 and was amended on (MM/DD/YYYY) _____ (if applicable).

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Prior Foreign Application Number(s)	Country	Foreign Filing Date (MM/DD/YYYY)	Priority Not Claimed	Certified Copy Attached? YES	Certified Copy Attached? NO
199 19 769.5	Germany	04/30/1999	☐	☐	☒

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U.S. Parent Application Number	PCT Parent Number	Parent Filing Date (MM/DD/YYYY)	Parent Patent Number *(if applicable)*
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Name of Sole or First Inventor: ☐ A petition has been filed for this unsigned inventor

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1-10

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ADDITIONAL INVENTOR(S) Supplemental Sheet

Name of Additional Joint Inventor, if any: ☐ A petition has been filed for this unsigned inventor

2-00

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Name of Additional Joint Inventor, if any: ☐ A petition has been filed for this unsigned inventor

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As a below named inventor, I hereby declare that:
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I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

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Name	Registration Number	Name	Registration Number
Wayne C. Jaeschke	21,062		
Glenn E. J. Murphy	33,539		
Stephen D. Harper	33,243		
Kimberly R. Hild	39,224		

☐ Additional attorney(s) and/or agent(s) named on a supplemental sheet attached hereto.

Please direct all correspondence to: ☒ Customer Number or label **00423** OR ☐ Fill in correspondence address below

Name	Kimberly R. Hild				
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I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Name of Sole or First Inventor: ☐ A petition has been filed for this unsigned inventor

Given Name	**Christine**	Middle Initial		Family Name	**Schroeder**	Suffix e.g. Jr.	
Inventor's Signature						Date	
Residence: City	**Duesseldorf**	State		Country	**Germany**	Citizenship	**Germany**
Post Office Address	**Am Alten Rhein 28**						
Post Office Address							
City	**40593 Duesseldorf**	State		Zip		Country	**Germany**	Applicant Authority	

☒ Additional inventors are being named on supplemental sheet(s) attached hereto

(+) inside this box →

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DECLARATION

ADDITIONAL INVENTOR(S) Supplemental Sheet

Name of Additional Joint Inventor, if any: ☐ A petition has been filed for this unsigned inventor

Given Name	Hans-Theo	Middle Initial		Family Name	Leinen	Suffix e.g. Jr.	
Inventor's Signature						Date	
Residence: City	Duesseldorf	State		Country	Germany	Citizenship	Germany
Post Office Address	Gertrudisstrasse 2						
Post Office Address							
City	40229 Duesseldorf	State		Zip		Country	Germany	Applicant Authority	

Name of Additional Joint Inventor, if any: ☐ A petition has been filed for this unsigned inventor

Given Name	Bernhard	Middle Initial		Family Name	Banowski	Suffix e.g. Jr.	
Inventor's Signature						Date	
Residence: City	Duesseldorf	State		Country	Germany	Citizenship	Germany
Post Office Address	Benrodestrasse 6						
Post Office Address							
City	40597 Duesseldorf	State		Zip		Country	Germany	Applicant Authority	

Name of Additional Joint Inventor, if any: ☐ A petition has been filed for this unsigned inventor

Given Name	Marcel	Middle Initial		Family Name	Roth	Suffix e.g. Jr.	
Inventor's Signature						Date	
Residence: City	Duesseldorf	State		Country	Germany	Citizenship	Germany
Post Office Address	Am Nettchesfeld 21						
Post Office Address							
City	40589 Duesseldorf	State		Zip		Country	Germany	Applicant Authority	

Name of Additional Joint Inventor, if any: ☐ A petition has been filed for this unsigned inventor

5-00

Given Name	Johann	Middle Initial		Family Name	Glasl	Suffix e.g. Jr.	
Inventor's Signature	Johann Glasl					Date	Nov. 6, 2001
Residence: City	Solingen	State		Country	Germany	Citizenship	Germany
Post Office Address	Becher Strasse 82						
Post Office Address							
City	42719 Solingen	State		Zip		Country	Germany	Applicant Authority	

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☐ Assignee of record of the entire interest. See 37 CFR 3.71.
Statement under 37 CFR 3.73(b) is enclosed. (Form PTO/SB/96).

☒ Attorney or agent of record.

Typed or Printed Name	Kimberly R. Hild, R.N. 39,224
Signature	Kimberly R. Hild
Date	June 6, 2002

NOTE: Signatures of all the inventors or assignees of record of the entire interest or their representative(s) are required. Submit multiple forms if more than one signature is required, see below*.

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